

Effects of amendments of paper mill sludge and nutrients on soil surface CO₂ flux in northern hardwood forests

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Abstract: Safe and economical disposal of paper mill sludge is a key consideration for forest products industry. A study was conducted to examine the effects of amendments of sludge and nutrients on soil surface CO₂ flux (R_S) in northern hardwood forests and to quantify the relationship among R_S, soil temperature, and moisture in these stands. The experiment was a randomized complete block design that included sludge-amended, fertilized, and control treatments in sugar maple (*Acer saccharum* Marsh) dominated hardwood forests in the Upper Peninsula of Michigan, USA. Results showed that R_S was positively correlated to soil temperature ($R^2 = 0.80$, $p < 0.001$), but was poorly correlated to soil moisture. Soil moisture positively affected the R_S only in the sludge-amended treatment. The R_S was significantly greater in the sludge-amended treatment than in the fertilized ($p = 0.033$) and the control ($p = 0.048$) treatments. The maximum R_S in the sludge-amended treatment was $8.8 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$, 91% and 126% greater than those in the fertilized ($4.6 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) and control ($3.9 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) treatments, respectively. The R_S did not differ significantly between the fertilized and control treatments. The difference in R_S between sludge-amended and the other treatments decreased with time following treatment.

Keywords: Paper mill sludge; Fertilization; Soil surface CO₂ flux; Environmental factor

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Introduction

The pulp and paper industry, a major component of the forestry-related economy in the USA, produces large quantities of wastewater treatment residuals – or paper mill sludge, which must be disposed in an environmentally safe manner. Approximately 5.3×10^6 t of paper mill sludge was produced in 1995 (NCASI 1999), much of which was disposed in landfills. Many of the existing landfills are near capacity, and the cost of creating new landfills has increased dramatically due to increased regulations affecting their planning and construction (Camberato *et al.* 1997). Therefore, it is a pressing concern for the pulp and paper industry to develop alternative methods for disposing paper mill sludge that reduce costs without harming the environment. One possible alternative to using landfills is the application of paper mill sludge to forests.

Paper mill sludge amendments provide large quantities of carbon and nutrients to forest soils and hence affect the decomposition and mineral cycles. The potential effects of applying paper mill sludge on the soil physical and chemical properties and leachates have been extensively investigated in forest ecosystems and agroecosystems (Bockheim *et al.* 1988; Kraske and Fernandez 1993; Simard *et al.* 1998; Chantigny *et al.* 1999; Xiao *et al.* 1999; Vance 2000), but the effects of sludge application on soil surface CO₂ flux (R_S), the second largest carbon flux in ter-

restrial ecosystems (Raich and Schlesinger 1992), are poorly understood.

The objectives of this study were to: (1) examine the effects of amendments of sludge and fertilizers on R_S in northern hardwood forests; and (2) quantify the relationship among R_S, soil temperature, and moisture in these stands. We hypothesized that application of sludge and fertilizers would increase R_S because increased nutrient availability could stimulate heterotrophic respiration (Foster *et al.* 1980).

Methods

Site description and experimental design

The study was conducted in the Upper Peninsula, near Norway, Michigan, USA (45°47'N, 87°45'W). The long-term (29 years) mean January and July air temperatures for this area are -11.0 °C and 19.8 °C, respectively. The mean annual temperature is 5.2 °C. Mean annual rainfall and snowfall are 739 and 1595 mm, respectively. The dominant soil is a mixed typical eutroboralf.

The forest is mesic northern hardwood, dominated (92% of the total density) by sugar maple. Other tree species, in order of importance, include American basswood (*Tilia americana* L.), white ash (*Fraxinus americana* L.), American beech (*Fagus grandifolia* Ehrh.), and black cherry (*Prunus serotina* Ehrh.). The understory consists of sugar maple seedlings, as well as assorted perennial herbs, with a relatively large presence of leek (*Allium tricoccum* Ait). General stand structure and soil characteristics were summarized in Table 1.

The experiment was a randomized complete block design with three treatments and three blocks. Three blocks were established in May 1997, two at the Waucedah (WAUC1 and WAUC2) and one at the Nora's Camp site (NORA2). The three treatments, paper mill sludge, fertilization, and control, were randomly assigned to one of three plots in each block. The plots at WAUC1 and WAUC2 were 25m x 25m, and those at NORA2 were 50m x

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50m. Larger plots were used at the NORA2 site because of larger diameter trees and lower tree density (Table 1).

Table 1. General stand structure and soil characteristics of the study sites

Characteristics	WAUC1	WAUC2	NORA2
Geographic location	45°47'N, 87°45'W	45°47'N, 87°45'W	45°47'N, 87°39'W
Elevation (m)	315	315	320
Slope (%)	< 5	15-20	5-10
Aspect	S	S	SW
Stand age (years)	55	55	60
Stand density (tree · hm ⁻²)	530 (65)	414 (155)	371 (32)
Basal area (m ² · hm ⁻²)	20.0 (1.5)	14.9 (1.8)	20.3 (1.2)
PAI (m ² · m ⁻²) ^b	3.6	2.7	2.5
Average DBH (cm)	25.6 (1.1)	28.4 (1.6)	28.5 (0.5)
Soil texture	fine sandy loam	fine sandy loam	fine sandy loam
SOM (%)	5.1 (0.3)	7.3 (0.4)	7.4 (0.7)
Soil pH	5.41 (0.13)	5.74 (0.09)	5.97 (0.18)
Total N (%)	0.16 (0.02)	0.08 (0.03)	0.33 (0.03)

a. The data inside parentheses are standard errors. WAUC1, WAUC2, and NORA2 stand for the research sites at Waucedah site 1, 2, and the Nora's Camp site, respectively.

b. PAI = plant area index (one-half plant area index calculated using a Li-Cor LAI-2000).

c. The values for soil are the mean of 9 plots x 4 samples per plot collected at 0-20 cm depth. SOM stands for soil organic matter.

Fertilized and Sludge-amended treatments

Nutrients were obtained from commercially available fertilizer. The "target" fertilizer application rate was to apply N : P : K : S : Mg : Ca at a ratio of 100 : 50 : 33 : 9 : 5 : 4 (Gower *et al.* 1992) with N application of 100 kg · hm⁻². However, Ca was applied at a slightly higher rate due to the composition of the Mg/Ca fertilizer (Gran-U-Lime) (Table 2).

Table 2. Nutrient application rates in the sludge and fertilization treatments.

Nutrient	Sludge (kg · hm ⁻²)	Fertilizer (kg · hm ⁻²)	Fertilizer Source
C	3200	20	
N	140	100	Urea (46-0-0)
P	30	33	Triple superphosphate (0-46-0)
K	5	50	(0-0-60)
S	50	9	Elemental sulfur fertilizer
Mg	30	5	Gran-U-Lime
Ca	770	6	Gran-U-Lime

The application rate for the paper mill sludge was intended to provide total N at a rate equal to that of the fertilized treatment (100 kg N · hm⁻²). Samples of paper mill sludge were collected from Champion International mill (Norway, Michigan) prior to the treatment to calculate the target application rates. However, chemical analysis of a paper mill sludge sample obtained immediately after application revealed that the N content of sludge amendment was greater than the initial sample (Table 3) and 39% higher than the fertilization treatment (Table 2). The sludge (at a rate of 11.5 t · hm⁻² on dry weight) and fertilizer were applied on June 17 and June 26 of 1997, respectively. The applica-

tion was done by hand to minimize plot disturbance and ensure a homogeneous distribution of sludge.

Table 3. Nutrient analysis of the paper mill sludge^a

Nutrient	Concentration	Nutrient	Concentration
C	28.03	B	12
N	1.22	Mn	869
P	0.25	Fe	3221
K	0.05	Cu	10
Ca	6.77	Al	9914
Mg	0.29	Na	1935
S	0.41	C/N	23
Zn	1.09		

Note: Nutrient concentrations of C, N, P, K, Ca, Mg, and S are in %, others (except C/N ratio) are in mg kg⁻¹.

Soil surface CO₂ flux measurement

Soil surface CO₂ flux was measured with a Li-Cor 6200 portable CO₂ infrared gas analyzer (Li-Cor Inc., Lincoln, NE, USA) equipped with a custom 15 cm diameter chamber. R_S was measured approximately every two weeks immediately after the treatments, from July 1997 to January 1998 for a total of eleven repeated measurement trials that included a large range of soil temperatures and encompassed a full growing season.

Thin-walled (3.2 mm) polyvinyl chloride (PVC) collars (15 cm inside diameter x 5 cm tall) were beveled on one end to reduce compaction and disturbance of the soil and forest floor upon placement. Eight collars were inserted 3 cm into the soil at random locations in each plot and left in the same locations throughout the study for continuity. R_S was measured continuously five times at each collar, with each measurement corresponding to a 5 $\mu\text{mol} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$ increase in CO₂ concentration inside the chamber. Wang *et al.* (2002) provide a complete description of the measurement procedures.

Soil temperature and moisture were measured near the collars while R_S was being measured. Soil temperature was measured at 2 cm (T₂) and 10 cm (T₁₀) depths using a digital long stem thermometer (model no. 15-078K, Fisher Scientific, Pittsburgh, PA). Soil samples were collected from 0-2 cm and 2-10 cm depths near the collars and weighed the fresh weight, and brought back to laboratory for soil moisture determination gravimetrically. All the soil samples were oven-dried at 70 °C to constant weight and weighed to the nearest 0.01 g.

Statistical analysis

Statistical analyses were conducted using SAS software version 8.0 (SAS Institute Inc. 2000). The mixed effect procedure (PROC MIXED) (Littell *et al.* 1996) was used for the repeated measuring analyses, assigning treatment, soil temperature (T₂ or T₁₀), soil moisture (W₂ or W₁₀), and all possible interactions as fixed effects. Block and collar were treated as random effects. Natural logarithmic transformation of R_S (Ln (R_S)) was needed to achieve homoscedasticity. A spatial power covariance matrix was chosen for the autocorrelation between repeated measurements based on SBC (Schwarz's Bayesian Criterion). Collars within treatment x block were used as the subject in the repeated measure analysis. A backward elimination procedure was performed to remove the insignificant ($\alpha > 0.05$) terms. An analysis of covariance (ANCOVA) was conducted to test the overall treatment effect on R_S by setting temperature and moisture as covariates. Added-variable plots (Cook and Weisberg 1999)

were used to provide a visual assessment of the effect of soil moisture versus temperature on R_S . An analysis of variance (ANOVA) was conducted to test the treatment effect on R_S at a specific measurement trial.

Results

Relationships among soil surface CO_2 flux, soil temperature and moisture

T -ratios of the coefficients in the model $\ln(R_S) \sim T_{10} + W_{10}$ were all greater than those in the model $\ln(R_S) \sim T_2 + W_2$ in spite of the similar variability. Therefore, we used T_{10} and W_{10} as the predictors of R_S in this study (The results for T_2 and W_2 was not shown). A contrast test ($\alpha = 0.05$) suggested that the same model of R_S against T_{10} and W_{10} was statistically sufficient for the fertilized and control treatments, but a model with significant different coefficients and terms was required for the sludge-amended treatment (Table 4).

Table 4. Models of soil surface CO_2 flux (R_S) for different treatments ^a

Coefficient	DF	Estimate	SE	t – ratio	P
A	18	-0.5719	0.188	-3.04	0.007
B	638	-0.3929	0.163	-2.41	0.016
C	15	0.1071	0.011	9.32	< 0.001
D	638	0.0253	0.006	4.42	< 0.001
E	654	0.0172	0.005	3.80	< 0.001

a. DF stands for degree of freedom. SE stands for standard error.

b. The models are: for the control and fertilized treatments: $\ln(R_S) = A + C * T_{10}$; for the sludge-amended treatment: $\ln(R_S) = (A + B) + (C + D) * T_{10} + E * W_{10}$; where T_{10} and W_{10} represent soil temperature and moisture at 10 cm depth, respectively. \ln is natural logarithm.

The $\ln(R_S)$ was positively correlated to soil temperature for all the treatments ($R^2 = 0.80$, $n = 84$, $p < 0.001$) (Figure 1a), but was not significantly correlated to soil moisture for the control and fertilization treatments ($p = 0.975$) (Figure 1b). The $\log(R_S)$ was positively correlated to soil moisture only for the sludge-amended treatment (Table 4 and Figure 1b).

Effects of treatments on soil surface CO_2 flux

The soil surface CO_2 flux for the sludge-amended treatment was significantly greater than in the fertilized ($p = 0.033$) and the control ($p = 0.048$) treatments (Figure 2). The maximum R_S for the sludge-amended treatment was $8.8 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$, 91% and 126% greater than those for the fertilized ($4.6 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) and control ($3.9 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) treatments, respectively. The R_S for the sludge-amended treatment averaged $2.86 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$, and was 38% and 47% greater than for the fertilized ($2.07 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) and the control ($1.94 \mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) treatments, respectively. However, the difference in R_S between the sludge-amended treatment and the others tended to decrease with the time following the treatments (Figure 3). The relative difference in R_S between the sludge-amended and the control treatments decreased from 116% on day 21 to 20% on day 150 following the treatment; and that between the sludge-amended and the fertilized treatments decreased from 82% to 12%, respectively. There was no significant difference in R_S between the control and the fertilized treatments

($p = 0.661$). Blocking also had no significant effect on R_S ($p = 0.187$).

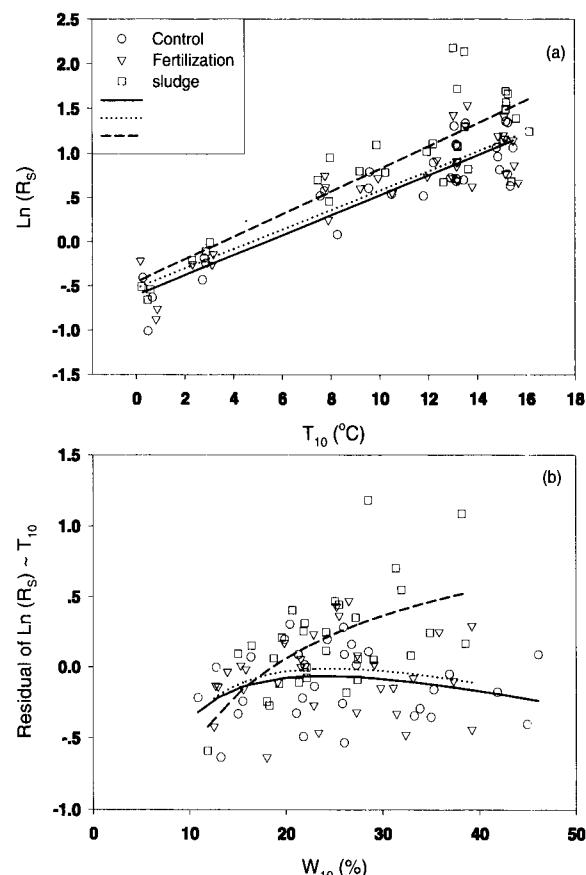


Fig. 1 – (a) The natural log of soil surface CO_2 flux (R_S , $\mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) versus soil temperature at 10 cm depth (T_{10} , $^{\circ}\text{C}$).

The overall statistics are: $R^2 = 0.80$, $n = 90$, $p < 0.001$. (b) The added-variable plot of $\ln(R_S)$ versus soil moisture at 10 cm depth (W_{10} , %) given the same soil temperature at 10 cm depth (T_{10} , $^{\circ}\text{C}$).

Discussion

Accurate estimates of soil surface CO_2 flux are important for constructing stand-level carbon budgets and assessing potential impacts of forest management on CO_2 exchange to the atmosphere, because R_S is the largest CO_2 flux from forest ecosystems to the atmosphere (Raich and Schlesinger 1992; Landsberg and Gower 1997). This flux is largely the sum of two processes: microbial respiration associated with decomposition of organic matter, and plant root respiration. A number of biophysical and biochemical factors affect the two processes differently and interactively.

Soil surface CO_2 flux is most strongly correlated to soil temperature (Raich and Schlesinger 1992; Lloyd and Taylor 1994; Wang *et al.* 2002). A temperature coefficient (Q_{10}) has commonly been used to estimate R_S (e.g. Raich and Schlesinger 1992). Our study also indicated that soil temperature alone explained 80% of the observed variations in the R_S . However, the Q_{10} model was unable to reflect the significant difference of the treatments on R_S measured in this study, because the models of R_S against temperature were statistically identical, i.e. had the similar intercepts ($p = 0.723$) and slopes ($p = 0.500$), for the three treatments (Figure 1a). Similar results about the inadequacy of a

single Q_{10} have been reported in other studies (Howard and Howard 1993; Lloyd and Taylor 1994; Rayment and Jarvis 2000; Wang *et al.* 2002).

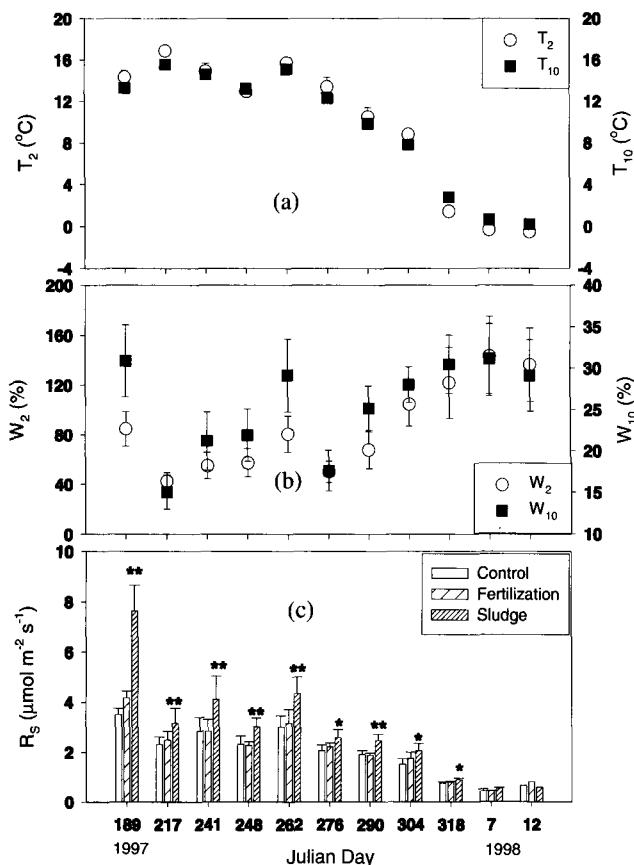


Fig. 2 Average soil surface CO_2 flux (R_s , $\mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$), soil temperature at 2 and 10 cm depth (T_2 , T_{10} , $^{\circ}\text{C}$), and corresponding moisture (W_2 , W_{10} , %) for the sludge-amended, fertilized, and control treatments on the specific sampling day

The error bars represent the standard errors of the three blocks. Missing standard errors on Day 12, 1998 is because soil samples were collected for only 1 block due to heavy snow. ** denotes that R_s for the sludge-amended treatment is significantly ($\alpha = 0.05$) greater than R_s for both fertilized and control treatments; * denotes that R_s for the sludge treatment is only significantly ($\alpha = 0.05$) greater than that for the control.

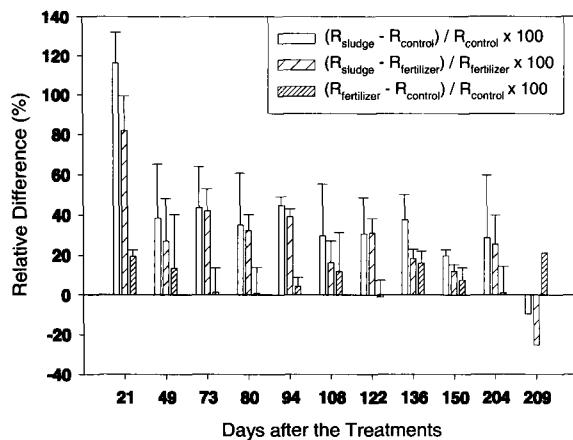


Fig. 3 Dynamics of relative differences in soil surface CO_2 flux (R_s , $\mu\text{mol CO}_2 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) among the treatments following the treatment. Bars represent block mean ($n = 3$) with the standard errors.

Soil moisture is another important environmental control on R_s , and often interactively influences R_s with soil temperature (Davidson *et al.* 1998; Orchard and Cook 1983; Wagai *et al.* 1998; Wang *et al.* 2002). However, this study showed that soil moisture did not significantly affect R_s in the fertilized and control treatments unless the moisture was below a critical value during the growing season (Figure 2b). When W_{10} was less than 18%, soil moisture exerted a strong limitation on R_s for all the three treatments. When the W_{10} was above this cutoff point, soil moisture showed little effect on R_s for the fertilized and control treatments. For the sludge-amended treatment, R_s was positively correlated to W_{10} for a broad range (observed from 12 to 38%). Although this study was not designed to differentiate the contribution of microbial respiration from autotrophic respiration, we did investigate the fine root biomass, the component responsible for the majority of autotrophic respiration (Landsberg and Gower 1997). Fine root net primary production estimated using in-growth core technique in 1999, two years after the treatments, was statistically similar for the three treatments (Feldkirchner 2000). We speculated that the R_s difference among the treatments might attribute to the difference in microbial respiration, which might have different sensitivity to soil moisture among the treatments.

Soil microbes responsible for decomposing organic matter are limited by the availability of carbon substrate (Foster *et al.* 1980), nitrogen (Jackman 1960), and soil acidity (Paul and Clark 1989). Paper mill sludge provided a labile carbon source with a low C/N ratio (Table 2) that could stimulate heterotrophic activity. We observed a substantial increase in R_s immediately after the sludge-amended treatment, and the difference in R_s between the sludge-amended and the other treatments declined with the time following the treatments (Figure 3). This suggested that the increase in R_s flux in the sludge-amended treatment was probably attributed to the carbon-enriched sludge, and declined as the labile carbon addition was used by heterotrophs, because microorganisms first colonize the surface of substrate and process the easily available carbon fraction at relatively rapid rate during the initial stage of decomposition (Ladd *et al.* 1996).

The addition of nitrogen increased soluble carbon and stimulated heterotrophic activity once microbial demand for carbon was met (Foster *et al.* 1980; Jackman 1960). Therefore, it seems reasonable that adding nutrients, especially nitrogen, would stimulate microbial activity, and hence increase R_s . Application of ammonium salts often decreased microbial respiration (Fessenden *et al.* 1971; Foster *et al.* 1980; Roberge 1976; Salomius 1972). Other studies have reported that R_s does not differ between control and fertilized forest soils (Castro *et al.* 1994; Fog 1988; Haynes and Gower 1995; Vose *et al.* 1995). One possible explanation for the similar R_s between the control and the fertilized treatments could be inadequate application rate of N, since no significant blocking effect in R_s was detected even though the total N concentration differed by four folds among the three blocks prior to the treatments (Table 1). Additionally, soil acidity could influence microbial respiration (Paul and Clark 1989). However, soil samples taken in the spring of 1998 indicated that soil pH was similar among the three treatments (Feldkirchner 2000).

In summary, soil temperature explained the majority of observed variation in the soil surface CO_2 flux, but was inadequate to describe the impact of sludge amendment on CO_2 flux unless soil moisture was included in the models. The significant increase in CO_2 exchange to the atmosphere due to sludge amend-

ment suggests that caution be taken when considering the application of paper mill sludge to the northern hardwood forests given concerns on the increase in atmospheric CO₂ concentration.

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